Table 1. *Atomic positional parameters and B_{eq} values for* (1), *with e.s.d.'s*

$$B_{eq} = (4/3)[a^2B_{11} + b^2B_{22} + c^2B_{33} + (2ab\cos\gamma)B_{12} + (2ac\cos\beta)B_{13} + (2bc\cos\alpha)B_{23}].$$

	x	*y*	*z*	B_{eq}(Å^2)
Br	0·28344 (7)	0·11981 (3)	0·07820 (6)	4·15 (1)
O	−0·2308 (5)	0·1133 (2)	−0·6789 (4)	4·58 (9)
N1	−0·3496 (5)	−0·0173 (2)	−0·6189 (5)	2·96 (9)
N2	−0·4606 (5)	−0·1322 (2)	−0·5533 (5)	3·5 (1)
C1	0·0105 (6)	0·0545 (3)	−0·2247 (5)	3·1 (1)
C2	0·1231 (6)	0·1160 (3)	−0·1620 (6)	3·1 (1)
C3	0·1201 (6)	0·1751 (3)	−0·2688 (7)	3·7 (1)
C4	0·0031 (6)	0·1739 (3)	−0·4422 (6)	3·7 (1)
C5	−0·1156 (6)	0·1123 (3)	−0·5095 (6)	3·2 (1)
C6	−0·1100 (6)	0·0524 (3)	−0·3981 (5)	2·7 (1)
C7	−0·2319 (6)	−0·0131 (3)	−0·4610 (5)	3·0 (1)
C8	−0·4661 (5)	−0·0817 (3)	−0·6724 (5)	2·9 (1)
C9	−0·5769 (6)	−0·0885 (3)	−0·8458 (6)	3·4 (1)
C10	−0·6871 (6)	−0·1520 (3)	−0·9028 (6)	3·7 (1)
C11	−0·6829 (6)	−0·2049 (3)	−0·7830 (7)	3·6 (1)
C12	−0·5697 (7)	−0·1925 (3)	−0·6117 (6)	3·9 (1)
H′O	−0·276 (5)	0·076 (3)	−0·697 (5)	4*

* Refined isotropically.

Table 2. *Selected bond distances* (Å) *and bond angles* (°) *with e.s.d.'s in parentheses*

O—C5	1·346 (5)	C3—C4	1·376 (6)
O—H′O	0·73 (5)	C4—C5	1·404 (7)
N1—C7	1·281 (5)	C5—C6	1·399 (7)
N1—C8	1·424 (6)	C6—C7	1·466 (6)
N2—C8	1·329 (6)	C8—C9	1·369 (6)
N2—C12	1·337 (6)	C9—C10	1·386 (7)
C1—C2	1·371 (6)	C10—H10	0·98 (4)
C1—C6	1·384 (5)	C11—C12	1·372 (6)
C2—C3	1·369 (7)		

C5—O—H′O	106 (3)	C1—C6—C7	118·7 (4)
C7—N1—C8	119·2 (4)	C5—C6—C7	121·2 (3)
C8—N2—C12	116·1 (4)	N1—C7—C6	121·6 (4)
C2—C1—C6	119·8 (4)	N1—C8—N2	118·8 (3)
C1—C2—C3	121·0 (4)	N1—C8—C9	117·7 (4)
C2—C3—C4	120·3 (4)	N2—C8—C9	123·5 (4)
C3—C4—C5	119·9 (5)	C8—C9—C10	119·0 (5)
O—C5—C4	118·6 (4)	C9—C10—C11	118·5 (4)
O—C5—C6	122·6 (4)	C10—C11—C12	118·2 (5)
C4—C5—C6	118·8 (4)	N2—C12—C11	124·6 (5)
C1—C6—C5	120·1 (4)		

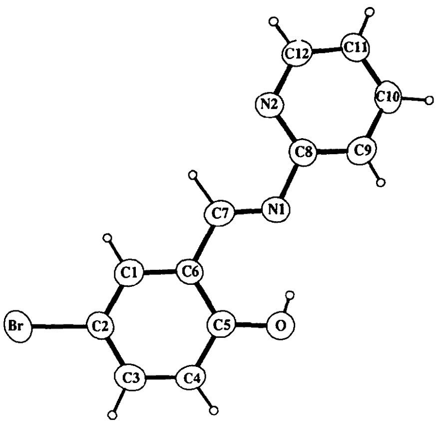

Fig. 1. Molecular structure of the title compound, with atom-numbering scheme.

1984). The 5-bromo derivative was synthetized because 2-(2-pyridyliminomethyl)phenol (salapy) did not give good crystals for X-ray diffraction studies.

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Structure of a Fully Protected Seco-erythronolide B Acid Derivative

By V. M. Lynch, W.-C. Lee, S. F. Martin and B. E. Davis

Department of Chemistry and Biochemistry, University of Texas at Austin, Austin, TX 78712, *USA*

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Abstract. (3*S*,4*R*,5*S*,6*R*,7*R*,9*R*,10*S*,11*S*,12*S*,13*S*,14*R*)-14-Benzyloxymethoxy-10,12-*O*-carbonyl-4-*N*-imidazolylcarbonyl-6,7-*O*-isopropylidene-3,5,7,9,11,13-hexamethylhexadec-1-ene-4,6,7,10,12,14-hexol, $C_{38}H_{56}N_2O_9$, M_r = 684·87, monoclinic, $P2_1$, a = 8·7302 (12), b = 15·597 (2), c = 14·463 (2) Å, β = 104·797 (10)°, V = 1904·1 (4) Å^3, Z = 2, D_x (198 K) = 1·19 g cm^{-3}, μ = 0·7893 cm^{-1}, Mo $K\alpha$ radiation,

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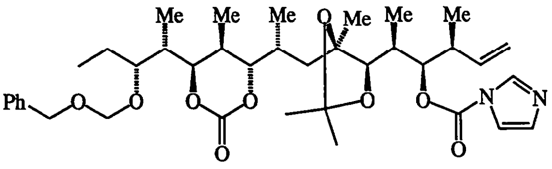
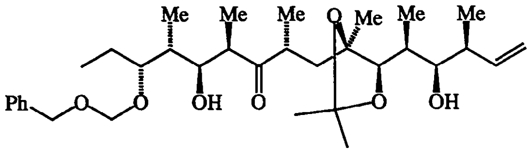
$\lambda = 0{\cdot}7107$ Å, $F(000) = 740$, $T = 198$ K, $R = 0{\cdot}0301$ for 3141 reflections $[F_o \geq 4\sigma(F_o)]$. The crystal structure was undertaken to determine the stereochemistry of the title compound. The molecule is folded on itself in such a way that the portion of the molecule from the phenyl ring at C44 and extending to C28 forms nearly a single turn of a right-handed screw. Large deviations from ideality for several Csp^3—Csp^3 bond angles are observed that are presumably due to intramolecular steric effects. The largest deviations are: C3—C4—C5 117·8 (2), C5—C6—C7 121·2 (2), C7—C8—C9 115·3 (2), C9—C10—C11 116·2 (2), C11—C12—C13 117·2 (2), C14—C15—C16 115·5 (3)°.

Experimental. Compound (1) was synthesized in two steps by the asymmetric reduction of (2) (Martin, Pacofsky, Gist & Lee, 1989) with tetramethylammonium triacetoxyborohydride $[Me_4NBH(OAc)_3]$ (Evans, Chapman & Carreira, 1988) followed by refluxing with 1,1′-carbonyldiimidazole in benzene.



(1)



(2)

Full details of the synthetic procedure will be described in due course (Martin & Lee, 1990). Crystals were obtained by slow evaporation of a hexane–diethyl ether solution. The data crystal was a clear, colorless plate of approximate dimensions 0·23 × 0·45 × 0·52 mm that was cut from a larger crystal. The data were collected on a Nicolet *R*3 diffractometer using a graphite monochromator and a Nicolet LT-2 low-temperature delivery system. Lattice parameters were obtained from the least-squares refinement of 40 reflections with $24{\cdot}4 < 2\theta < 28{\cdot}0°$. The data were collected using the ω-scan technique with a 2θ range 4·0–50·0°, with a 1·2° ω scan at 6–12° min^{-1} (h $-10\rightarrow10$, k $0\rightarrow18$, l $-17\rightarrow17$). A total of 7035 reflections were collected of which 3520 were unique ($R_{\rm int} = 0{\cdot}0143$ for averaging symmetry-equivalent reflections). Four reflections ($\bar{1}43$, $12\bar{4}$, $14\bar{2}$, $\bar{1}\bar{1}5$) were remeasured every 196 reflections to monitor instrument and crystal stability. A smoothed curve of the intensities of these check reflections was used to scale the data. The scaling factor ranged from 0·966–1·03. The data were also corrected for Lp effects but not absorption. The data reduction and decay correction were applied using the Nicolet XRD *SHELXTL-Plus* software package (Sheldrick, 1988). Reflections having $F_o < 4\sigma(F_o)$ were considered unobserved (379 reflections). The structure was solved by direct methods (Sheldrick, 1988) and refined by full-matrix least squares (Sheldrick, 1976). In all, 666 parameters were refined in blocks of 325 and 342 parameters with the scale factor refined in each block. The non-H atoms were refined with anisotropic thermal parameters. All H-atom positions were obtained from a ΔF map and refined with

Table 1. *Fractional coordinates and equivalent isotropic thermal parameters* (Å^2) *for the non-hydrogen atoms of* (1)

For anisotropic atoms, the U value is $U_{\rm eq}$, calculated as $U_{\rm eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{A}_{ij}$ where $\mathbf{A}_{ij}$ is the dot product of the ith and jth direct-space unit-cell vectors.

	x	y	z	U
C1	0·7245 (4)	1·0995 (2)	0·6967 (2)	0·0622 (12)
C2	0·6189 (3)	1·0930 (2)	0·7462 (2)	0·0479 (9)
C3	0·6493 (3)	1·0665 (2)	0·8498 (2)	0·0399 (8)
C4	0·5670 (3)	0·98045 (15)	0·85469 (15)	0·0311 (7)
C5	0·3869 (3)	0·97592 (14)	0·8122 (2)	0·0333 (7)
C6	0·3339 (3)	0·88264 (14)	0·81719 (14)	0·0316 (7)
C7	0·1801 (2)	0·84912 (13)	0·74952 (14)	0·0309 (7)
C8	0·2059 (3)	0·81927 (15)	0·65442 (15)	0·0309 (7)
C9	0·0598 (2)	0·77845 (15)	0·58457 (14)	0·0331 (7)
C10	0·1078 (3)	0·69390 (15)	0·5465 (2)	0·0370 (7)
C11	0·1484 (3)	0·62101 (14)	0·6177 (2)	0·0338 (7)
C12	−0·0044 (2)	0·58969 (15)	0·63804 (15)	0·0334 (7)
C13	0·0075 (3)	0·51398 (15)	0·7064 (3)	0·0368 (8)
C14	−0·1509 (3)	0·4957 (2)	0·7296 (2)	0·0450 (9)
C15	−0·1563 (4)	0·4115 (2)	0·7820 (2)	0·0598 (12)
C16	−0·1338 (4)	0·3322 (2)	0·7287 (3)	0·0671 (13)
C17	0·8248 (3)	1·0613 (2)	0·9009 (2)	0·0503 (9)
O18	0·5960 (2)	0·95666 (10)	0·95660 (10)	0·0351 (5)
C19	0·6586 (3)	0·8802 (2)	0·98143 (15)	0·0364 (7)
O20	0·7221 (2)	0·83355 (12)	0·93705 (12)	0·0518 (6)
N21	0·6438 (2)	0·85876 (13)	1·07328 (12)	0·0388 (6)
C22	0·5445 (3)	0·8942 (2)	1·1221 (2)	0·0515 (10)
N23	0·5572 (3)	0·8567 (2)	1·2042 (2)	0·0676 (10)
C24	0·6699 (4)	0·7931 (2)	1·2083 (2)	0·0665 (12)
C25	0·7235 (3)	0·7926 (2)	1·1295 (2)	0·0541 (10)
C26	0·2991 (3)	1·0416 (2)	0·8585 (2)	0·0477 (9)
O27	0·3095 (2)	0·86427 (10)	0·90943 (10)	0·0416 (5)
C28	0·2346 (3)	0·7823 (2)	0·9014 (2)	0·0432 (8)
O29	0·1537 (2)	0·77310 (11)	0·80187 (10)	0·0387 (5)
C30	0·1169 (5)	0·7827 (3)	0·9618 (2)	0·0689 (13)
C31	0·3594 (4)	0·7127 (2)	0·9293 (2)	0·0552 (10)
C32	0·0388 (3)	0·9086 (2)	0·7375 (2)	0·0452 (9)
C33	−0·0133 (4)	0·8409 (2)	0·5039 (2)	0·0559 (10)
O34	−0·0186 (2)	0·66622 (12)	0·46321 (10)	0·0496 (6)
C35	−0·1194 (3)	0·6030 (2)	0·4659 (2)	0·0440 (8)
O36	−0·1129 (2)	0·56202 (11)	0·54783 (11)	0·0437 (5)
O37	−0·2161 (2)	0·58237 (13)	0·39407 (12)	0·0602 (7)
C38	0·2414 (3)	0·5508 (2)	0·5827 (2)	0·0496 (10)
C39	0·1364 (4)	0·5313 (2)	0·7978 (2)	0·0524 (10)
O40	−0·1779 (2)	0·56568 (13)	0·78912 (11)	0·0535 (6)
C41	−0·3380 (4)	0·5866 (3)	0·7785 (3)	0·0670 (13)
O42	−0·3977 (2)	0·63977 (13)	0·69905 (14)	0·0610 (7)
C43	−0·3335 (3)	0·7243 (2)	0·7103 (2)	0·0503 (10)
C44	−0·3847 (2)	0·7710 (2)	0·6170 (2)	0·0395 (8)
C45	−0·4103 (3)	0·7283 (2)	0·5300 (2)	0·0488 (10)
C46	−0·4539 (3)	0·7735 (2)	0·4449 (2)	0·0560 (10)
C47	−0·4731 (3)	0·8611 (2)	0·4448 (2)	0·0558 (10)
C48	−0·4496 (3)	0·9041 (2)	0·5305 (2)	0·0509 (10)
C49	−0·4054 (3)	0·8594 (2)	0·6149 (2)	0·0434 (9)

Table 2. *Bond lengths* (Å) *and angles* (°) *for the non-hydrogen atoms of* (1)

1	2	3	1—2	1—2—3
C2	C1		1·308 (5)	
C3	C2	C1	1·511 (4)	126·4 (3)
C4	C3	C17	1·532 (3)	110·9 (2)
C4	C3	C2		108·6 (2)
C17	C3	C2	1·524 (3)	113·3 (2)
C5	C4	O18	1·536 (3)	106·6 (2)
C5	C4	C3		117·8 (2)
O18	C4	C3	1·478 (3)	107·6 (2)
C6	C5	C26	1·534 (3)	115·0 (2)
C6	C5	C4		108·3 (2)
C26	C5	C4	1·532 (4)	111·7 (2)
C7	C6	O27	1·537 (3)	102·2 (2)
C7	C6	C5		121·2 (2)
O27	C6	C5	1·433 (3)	110·6 (2)
C8	C7	O29	1·523 (3)	107·5 (2)
C8	C7	C32		112·8 (2)
C8	C7	C6		111·8 (2)
O29	C7	C32	1·456 (3)	109·0 (2)
O29	C7	C6		100·03 (15)
C32	C7	C6	1·517 (3)	114·6 (2)
C9	C8	C7	1·548 (3)	115·3 (2)
C10	C9	C33	1·528 (3)	112·0 (2)
C10	C9	C8		109·5 (2)
C33	C9	C8	1·528 (4)	110·5 (2)
C11	C10	O34	1·514 (3)	109·4 (2)
C11	C10	C9		116·2 (2)
O34	C10	C9	1·475 (3)	109·3 (2)
C12	C11	C38	1·518 (3)	113·7 (2)
C12	C11	C10		108·0 (2)
C38	C11	C10	1·525 (4)	111·5 (2)
C13	C12	O36	1·527 (3)	105·7 (2)
C13	C12	C11		117·2 (2)
O36	C12	C11	1·468 (2)	108·8 (2)
C14	C13	C39	1·531 (4)	110·5 (2)
C14	C13	C12		111·7 (2)
C39	C13	C12	1·525 (3)	109·7 (2)
C15	C14	O40	1·523 (4)	108·9 (2)
C15	C14	C13		114·5 (2)
O40	C14	C13	1·446 (3)	106·5 (2)
C16	C15	C14	1·496 (5)	115·5 (3)
C19	O18	C4	1·323 (3)	116·8 (2)
O20	C19	N21	1·195 (3)	122·0 (2)
O20	C19	O18		128·2 (2)
N21	C19	O18	1·407 (3)	109·8 (2)
C22	N21	C25	1·368 (4)	106·7 (2)
C22	N21	C19		127·9 (2)
C25	N21	C19	1·386 (3)	125·3 (2)
N23	C22	N21	1·303 (4)	111·8 (3)
C24	N23	C22	1·388 (5)	104·7 (3)
C25	C24	N23	1·339 (5)	111·5 (3)
N21	C25	C24		105·3 (3)
C28	O27	C6	1·428 (3)	106·1 (2)
O29	C28	C30	1·440 (2)	110·3 (2)
O29	C28	C31		109·3 (2)
O29	C28	O27		105·5 (2)
C30	C28	C31	1·509 (5)	113·5 (3)
C30	C28	O27		108·3 (2)
C31	C28	O27	1·518 (4)	109·6 (2)
C7	O29	C28		109·7 (2)
C35	O34	C10	1·329 (3)	123·8 (2)
O36	C35	O37	1·335 (3)	120·0 (2)
O36	C35	O34		120·2 (2)
O37	C35	O34	1·203 (3)	119·8 (2)
C12	O36	C35		121·3 (2)
C41	O40	C14	1·405 (4)	114·8 (2)
O42	C41	O40	1·405 (4)	112·2 (3)
C43	O42	C41	1·425 (4)	113·5 (2)
C44	C43	O42	1·498 (4)	109·2 (2)
C45	C44	C49	1·391 (4)	117·7 (2)
C45	C44	C43		121·6 (2)
C49	C44	C43	1·390 (4)	120·7 (2)
C46	C45	C44	1·386 (4)	120·3 (3)
C47	C46	C45	1·377 (5)	120·7 (3)
C48	C47	C46	1·378 (4)	119·5 (3)
C49	C48	C47	1·372 (4)	119·7 (3)
C44	C49	C48		122·0 (2)

isotropic thermal parameters. The atom positions in the block not being refined served to define the origin along the screw axis. The function $\sum w(|F_o| - |F_c|)^2$ was minimized, where $w = 1/[\sigma(F_o)]^2$ and $\sigma(F_o) =$ $(0{\cdot}5kI^{-1/2}\{[\sigma(I)]^2 + (0{\cdot}02I)^2\}^{1/2})$. The intensity, I, is given by $(I_{\text{peak}} - I_{\text{backgound}}) \times$ (scan rate); 0·02 is a factor to downweight intense reflections and to account for instrument instability and k is the correction due to Lp effects and decay. $\sigma(I)$ was estimated from counting statistics; $\sigma(I) = [(I_{\text{peak}} + I_{\text{background}})^{1/2} \times \text{(scan rate)}]$. The final $R = 0{\cdot}0301$ for 3141 reflections, with $wR = 0{\cdot}0328$ ($R_{\text{all}} = 0{\cdot}0357$, $wR_{\text{all}} = 0{\cdot}0344$) and $S = 1{\cdot}140$. The maximum $|\Delta/\sigma| < 0{\cdot}1$ in the final refinement cycle and the minimum and maximum peaks in the final ΔF map were $-0{\cdot}19$ and $0{\cdot}14$ e Å^{-3}, respectively. The scattering factors for the non-H atoms were taken from Cromer & Mann (1968), with the anomalous-dispersion corrections taken from the work of Cromer & Liberman (1970). The scattering factors for the H atoms were obtained from Stewart, Davidson & Simpson (1965). Values used to calculate the linear absorption coefficient are from *International Tables for X-ray Crystallography* (1974, Vol. IV, p. 55).* Figures were generated using *SHELXTL-Plus* (Sheldrick, 1988). The positional and thermal parameters for non-H atoms are listed in Table 1, while the bond lengths and angles for the non-H atoms are listed in Table 2. The atom-labeling scheme is shown in Fig. 1. Other computer programs used in this work are listed in reference 11 of Gadol & Davis (1982).

Related literature. The asymmetric synthesis of the seco acid of erythronolide B has been recently

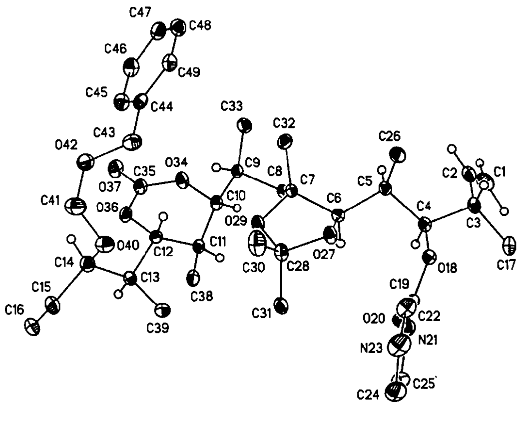


Fig. 1. View of (1) showing the atom-labeling scheme. Only the H atoms of the ethene moiety and those at chiral centers are shown. The non-H atoms are scaled to the 30% probability level while the H atoms are drawn to an arbitrary size.

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* Lists of anisotropic thermal parameters, H-atom positional parameters, bond distances and angles involving H atoms, torsion angles, structure-factor amplitudes and a unit-cell packing diagram have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53689 (37 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

reported (Martin *et al.*, 1989, and references therein). The crystal structure of a precursor of (1) and (2) has also been reported (Lynch, Pacofsky, Martin & Davis, 1989).

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Structure of 6-(4-*n*-Propoxybenzoyloxy)flavone

BY TAKAHIRO KAWAI, YUKIO YOSHIMURA AND KOHJI SHIMAOKA

Department of Physics, Faculty of Science and Engineering, Ritsumeikan University, Tojiin-kitamachi,
Kita-ku, Kyoto 603, *Japan*

NAOTAKE NAKAMURA

Department of Chemistry, Faculty of Science and Engineering, Ritsumeikan University, Tojiin-kitamachi,
Kita-ku, Kyoto 603, *Japan*

AND SHINICHI YANO

Department of Chemistry, Faculty of Engineering, Gifu University, Yanagido, Gifu 501-11, *Japan*

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Abstract. 4-Oxo-2-phenyl-4*H*-1-benzopyran-6-yl 4-propoxybenzoate, $C_{25}H_{20}O_5$, M_r = 400·43, triclinic, $P\bar{1}$, a = 16·306 (11), b = 14·830 (12), c = 13·918 (20) Å, α = 107·24 (9), β = 99·80 (9), γ = 72·33 (6)°, V = 3050 (5) Å^3, Z = 6, D_x = 1·306 g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, μ = 0·849 cm^{-1}, F(000) = 1260, T = 298 K, R = 0·076, wR = 0·047 for 10 683 unique observed reflections. There are no unusual bond distances or angles. The flavone skeleton is twisted with respect to the 4-*n*-propoxybenzoyloxy group in the molecule.

Experimental. The title compound, 6-(4-*n*-propoxybenzoyloxy)flavone, was prepared by a conventional dehydrochloride reaction of 6-hydroxyflavone and 4-*n*-propoxybenzoyl chloride in a mixture of benzene and pyridine (Hirose, Tsuya, Nishigaki, Idaka & Yano, 1989) and purified by recrystallization from acetone. A single crystal was obtained as a transparent and elongated rod by slow evaporation from acetone solution. A crystal with approximate dimensions 0·5 × 0·5 × 0·5 mm was mounted on a Rigaku ROTA AFC-5 diffractometer equipped with a graphite monochromator. The lattice parameters were obtained from the setting angles of 25 reflections with $5 < 2\theta < 10°$, Mo $K\alpha$ radiation. Intensities were measured within the ranges $0 < 2\theta < 65°$, $-20 \le h \le 20$, $-20 \le k \le 20$, $0 \le l \le 15$, using ω–2θ scans. Intensities of three standard reflections (520, $42\bar{4}$, 624) were measured every 100 reflections (maximum correction on I was < 1·8%). Lorentz and polarization corrections but no absorption correction were applied. 10 683 unique observed reflections were measured with $F_o \ge 2\sigma(F_o)$. The structure was determined by direct methods using *RANTAN* (Yao, 1981). The H-atom positions were geometrically obtained, non-H atoms refined with anisotropic thermal parameters. Final refinement including H atoms was made by full-matrix least-squares minimization using *UNICS* (Sakurai, 1967). Atomic scattering factors are from *International Tables for X-ray Crystallography* (1974, Vol. IV). The function $\sum w(F_o - F_c)^2$ was minimized, where $w = 1/\sigma(F_o)^2$. Final R = 0·076, wR = 0·047, S = 5·41,

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